# Method Validation for Selecting Baseline Solvent

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## Outline

- Introduction & Background
- Objective
- Experimental Method
- Results & Discussion
- Conclusion

# Introduction & Background

# WSTF's Experience

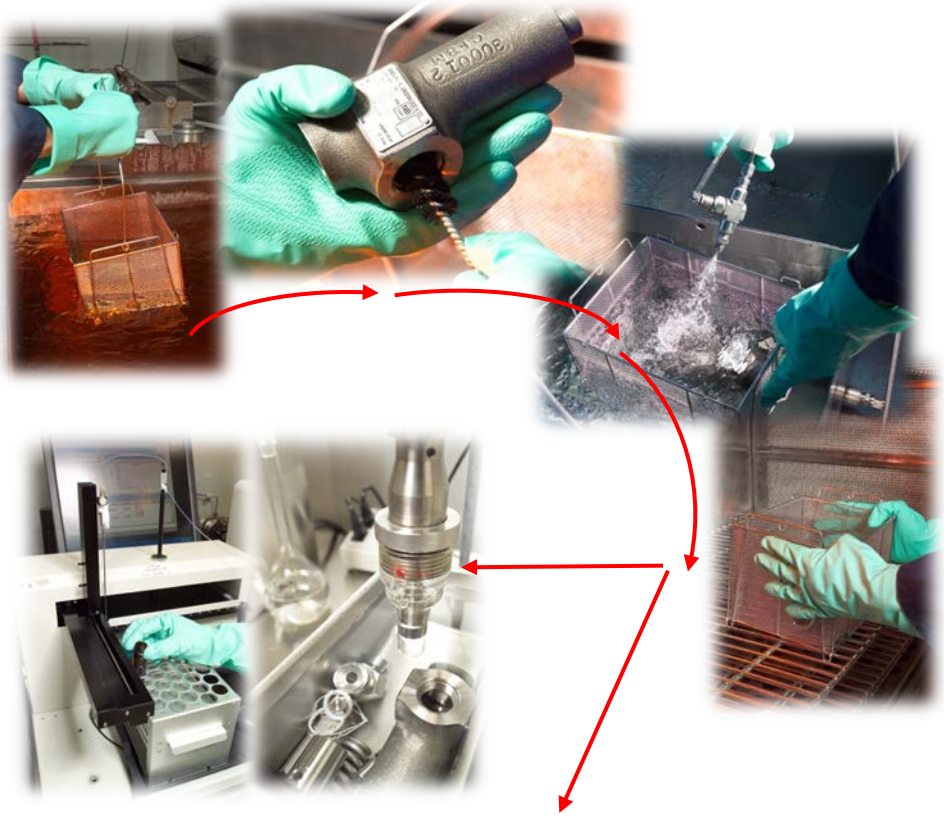
**Replacing CFC-113 in the 90's**

- Collaboration
  - DOD
  - NASA
    - Contractors
  - Solvent Manufactures
- Solvents
  - CFC
  - HCFC
  - HFE
  - TCE

- Considerations for Solvents
  - Cleaning effectiveness
  - $O_2$ compatibility
  - Materials compatibility
  - Aerospace fluids compatibility

## WSTF Cleaning Process

- Pre-cleaning
  - Ultra sonic
- Visual inspection
- Final clean/ cleanliness verification
  - DI water
  - HFE 7100
  - IPA
- Process Validation
  - AK 225G (baseline solvent)



# Problem Statement

- WSTF Processes is dependent on AK-225G for process validation
- AK-225G limited availability in 2015

# Objective

- To establish a standardized method for the evaluation of suitable replacement *baseline* solvent AK-225G.

# Experimental Method

# Standards for Testing Solvents

- ASTM G121. *Standard Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents*. ASTM International, West Conshohocken, Pennsylvania, 1998.
- ASTM G122. *Standard Test Method for Evaluating the Effectiveness for Cleaning Agents*. ASTM International, West Conshohocken, Pennsylvania, 1996 Revised 2008.
- ASTM G127. *Standard Guide for the Selection of Cleaning Agents for Oxygen Systems.* ASTM International, West Conshohocken, Pennsylvania, 1995 revised 2008.

# Attributes for a Baseline Solvent

## ASTM Considerations

- Toxicity
- Carcinogenicity
- Recyclability
- Waste Disposal
- Ozone Depletion
- Inertness (Flammability and combustibility)
  - Oxygen compatibility
- Availability and technical support from supplier
- Corrosivity & material compatibility
- Cost effectiveness
- Compliance with local, state, and federal regulations
- Application and use of Solvent

## Other Considerations

- HAP (Hazardous Air Pollutants)
- VOC (Volatile Organic Compounds)
- ACS reagent grade chemicals or higher
- Cleaning effectiveness
- Evaporation rate

# So Many Choices!

## ASTM Test Method Options

- Contamination of Coupons
  - Slurry
    - Contaminants: 1 vs. mixture
    - Concentration: 1 to 100 mg/mL
  - Application
    - 1 side vs. 2 sides
    - Pipette
    - Brush
    - Spray
    - Dip
  - Dry
    - Hang vs. laying flat
- Cleaning of coupons
  - Manufacture's recommended use of solvent
  - Sonication
  - Elevated  temperatures

# Experiment Outline

## Steps

1. Clean the coupons (standard)
2. Weigh the coupons (Tare weight)
3. Contaminate coupons
   a) Analyze slurry filter/NVR
   b) Oven dried
4. Weigh the coupon (Determine Contaminant)
5. Clean the coupon (test solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried
6. Weigh the coupon (Residual contaminant)
7. Verify cleanliness of coupon (AK-225-G, Verification solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried

## Matrix

- Series 1
  - 1 Contamination Slurry for each solvent
  - 3 solvents tested
  - 7 coupons for each solvent
- Series 2
  - 1 contamination Slurry
  - 6 solvents
  - 1 coupon for each solvent

## Contaminants

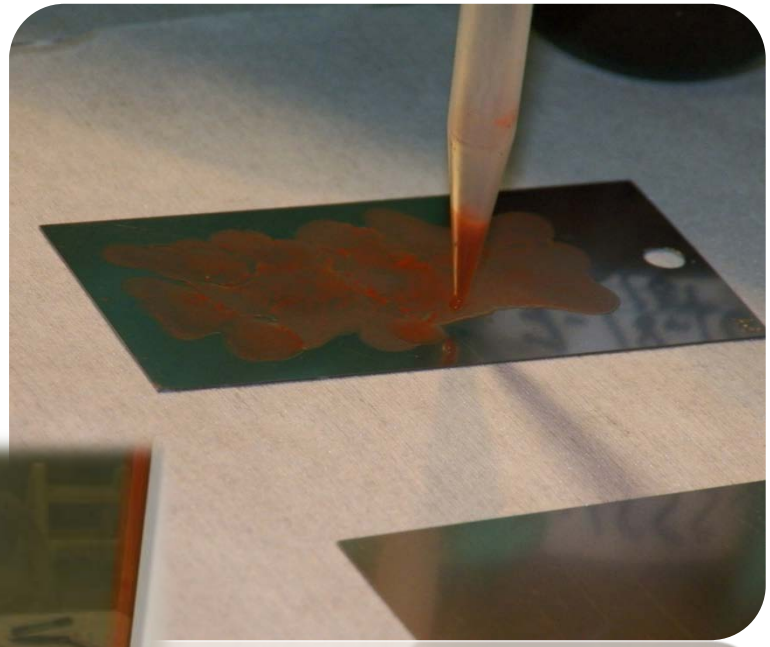
- Pump /hydrocarbon oil
- Hydraulic oil
- $O_2$ system lubricant
- Gage fluid
- Silicone  grease
- Dye penetrates- particles
- Iron powder 60 Mesh- particles

# Preparation of Contamination Slurry

- 5 slurry mixtures prepared
- 1 gram (+-.1g)
- 100 mL AK225-G
- Ultra Sonic 10 min
- Concentration 70 mg/mL

## Contamination of Coupons

- 600 µL contamination Slurry
- Bake
  - 1 hr.
  - 45 ºC (+/- 5ºC)
  - Nitrogen purge
- Cool off 1 hr.
- Weighed

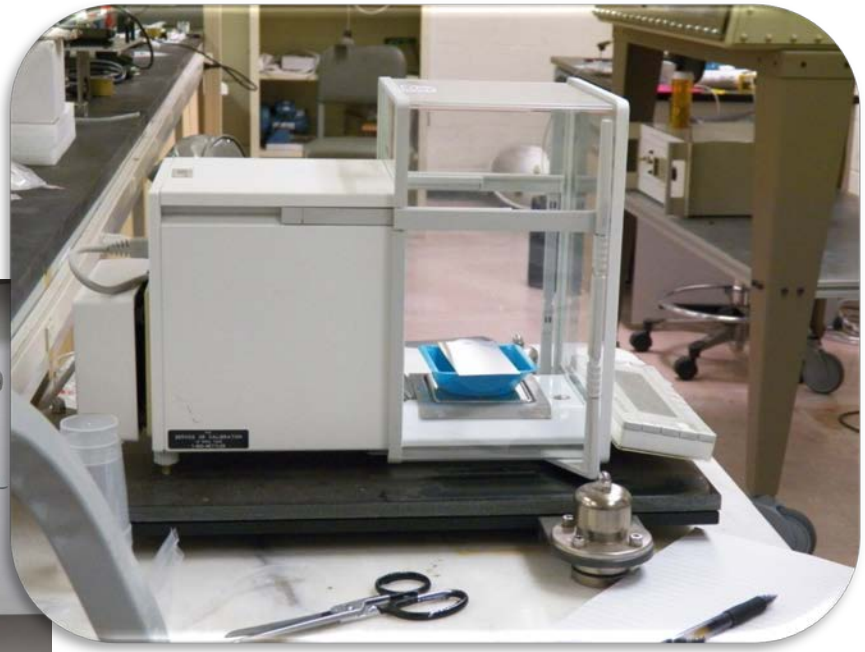


# Cleaning & Verification

- Flush with 100 mL of solvent
  - NVR analysis
  - Particle count
- Cleaned with test solvent
- Verified clean with AK225G

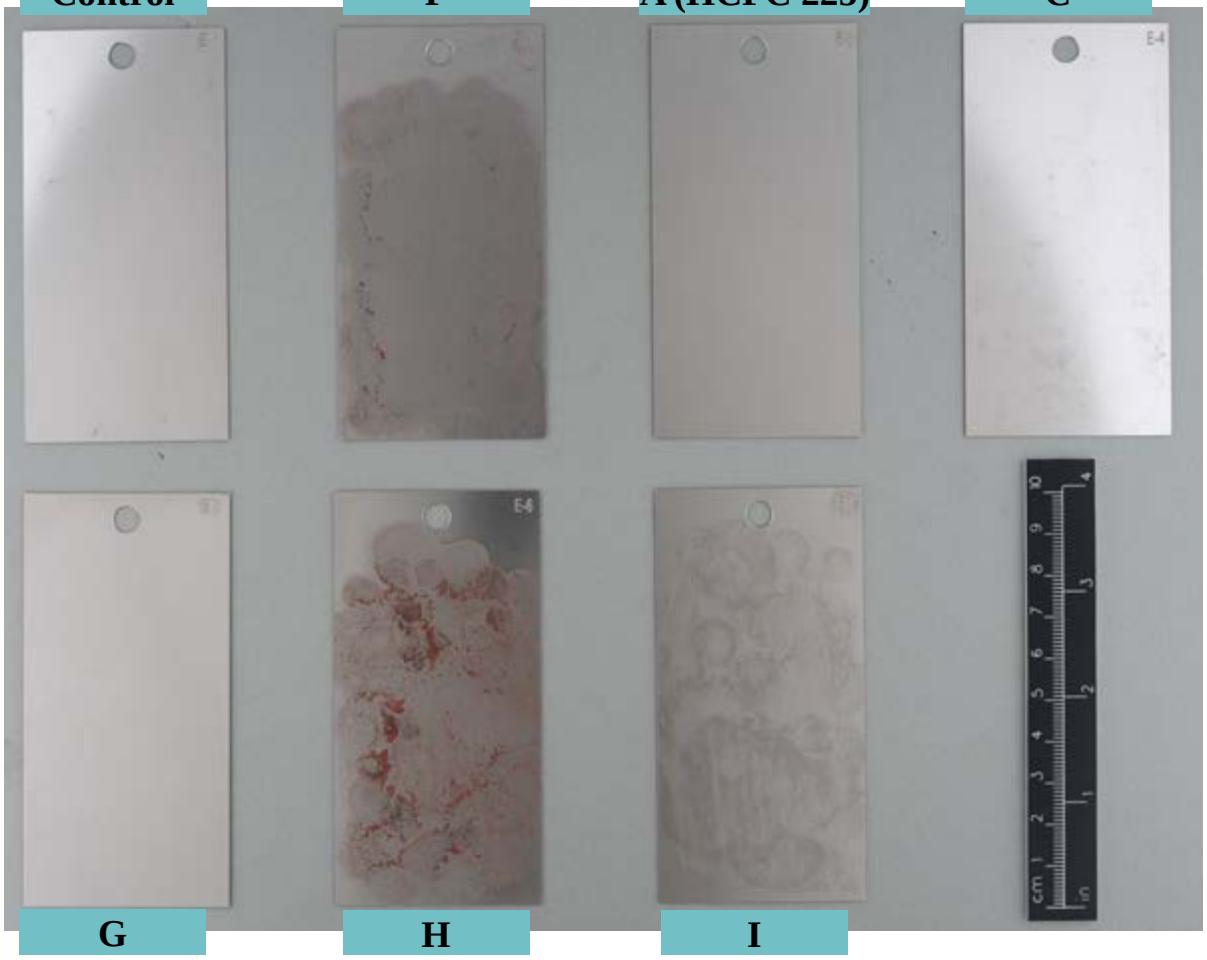
# Data Collection

- Counting Particles
- Weighing coupons
- Weighing NVR
- Weighing filters



# Results & Discussion

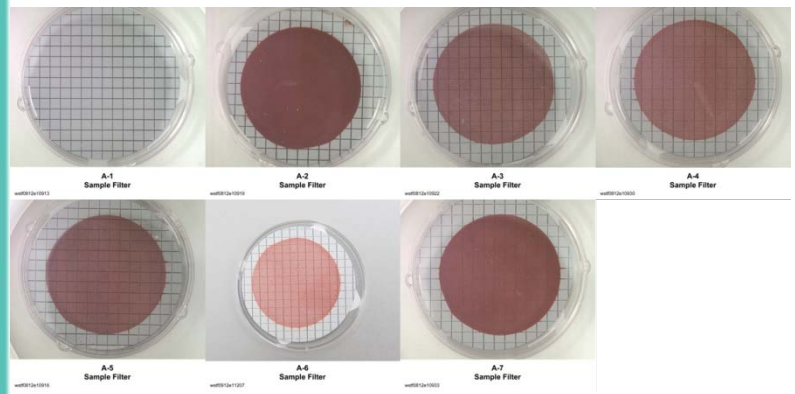
# Post Cleaned, Pre AK225G Validated Coupons



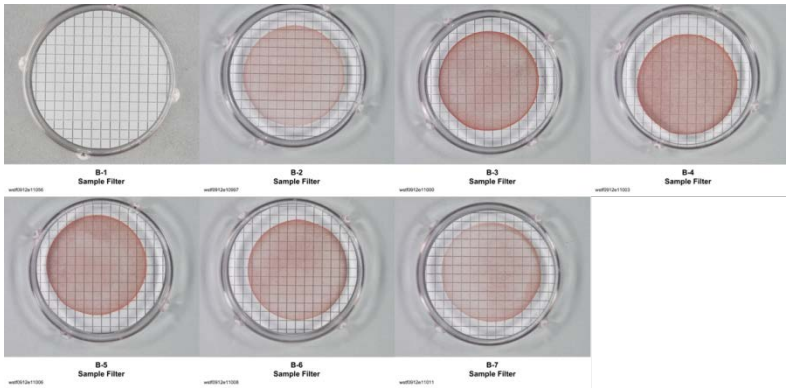

| Symbol | Description | Values |
|---|---|---|
| | **General** | |
| S | Surface Area (cm^2) | 45 |
| Ra | Surface Roughness (µm) | 17 |
| Spec. Bal | >10g-205g (g) | 0.002 |
| Clean Bal | 0 - 10 mg (+-g) | 0.00005 |
| Clean Bal | > 10 mg - 1 g (+-g) | 0.0001 |

# Post Cleaned, Pre Verification Filter Papers

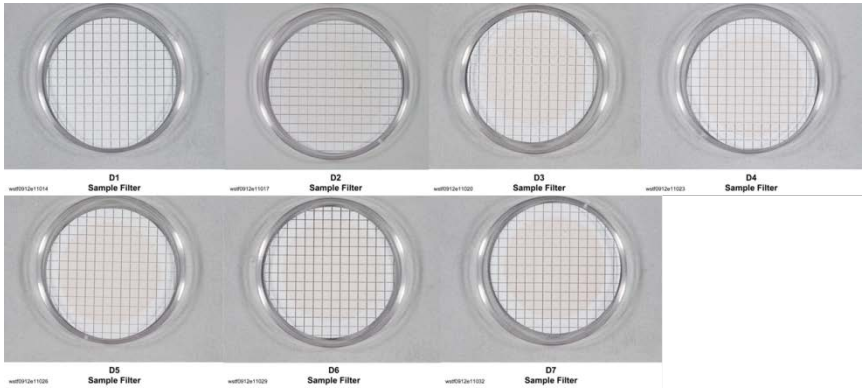
## Cleaned with AK-225G




## Cleaned with CFC-113

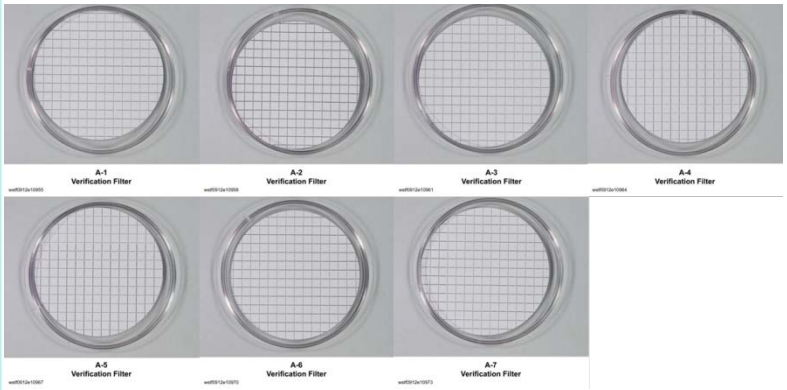



## Cleaned with D

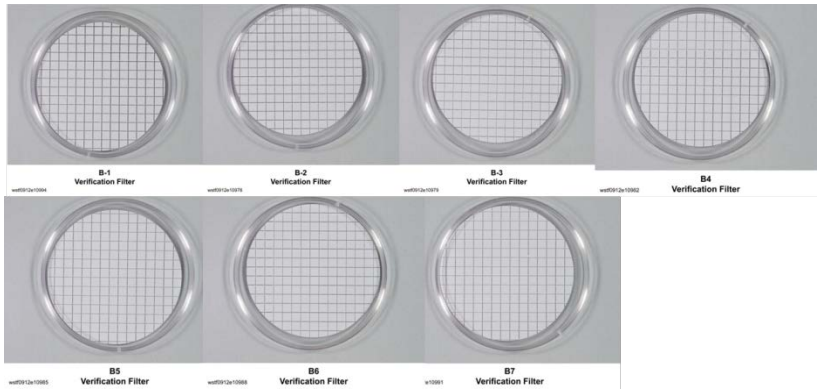


# Post From AK-225G *Verification* Filter Papers

## Cleaned with AK-225G




## Cleaned with CFC-113

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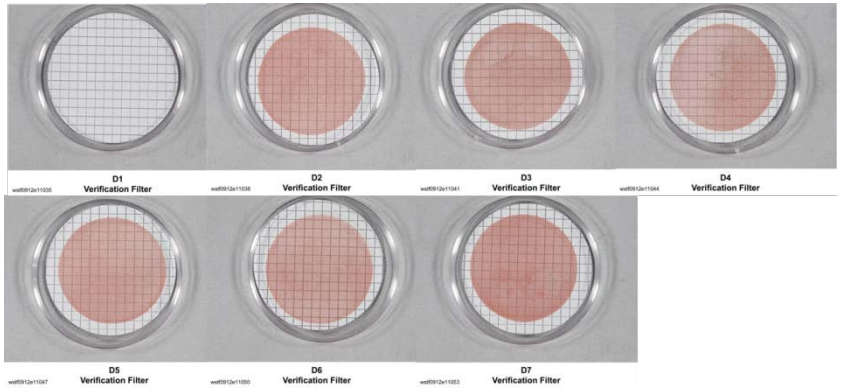

# Cleaning Efficiency of Solvents

**Comparison of Contamination Analyzed on Coupons**

| Symbol | Description | Solvents | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | *A {AK225G} (average) | *B {CFC} (average) | *D (average) | **F | **A {HCFC} | **C | **G | **H | **I |
| | Validation of experiment (g) | 0.0007 | -0.0002 | 0.0001 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 | 0.0004 |
| MCA | Mass of Contaminant Applied (g) | 0.0423 | 0.0316 | 0.0359 | 0.0450 | 0.0318 | 0.0335 | 0.0334 | 0.0526 | 0.0382 |
| MCR | Mass of Contaminant Removed (g) | 0.0410 | 0.0312 | 0.0127 | 0.0303 | 0.0308 | 0.0323 | 0.0325 | 0.0296 | 0.0355 |
| CEF | Cleaning Effectiveness Factor (%) | 97.11% | 98.88% | 35.68% | 67.33% | 96.86% | 96.42% | 97.31% | 56.27% | 92.93% |
| C | Amount of Contamination (mg/cm^2) | 0.9393 | 0.7015 | 0.7970 | 1.0000 | 0.7067 | 0.7444 | 0.7422 | 1.1689 | 0.8489 |
| RC | Residual Contamination (mg/cm^2) | 0.0270 | 0.0078 | 0.5152 | 0.3267 | 0.0222 | 0.0267 | 0.0200 | 0.5111 | 0.0600 |
| VNVR | Verification Nonvolatile Residue Weight | 0.0002 | 0.0005 | 0.0150 | | | | | | |

**Comparison of Removed Contamination Analyzed**

| Symbol | Description | Solvents | | |
|---|---|---|---|---|
| | | *AK-225G (average) | *CFC-113 (average) | *D (average) |
| SFW | Sample Filter contamination Weight (g) | 0.0189 | 0.0054 | 0.7970 |
| VFW | Verification Filter Contamination Weight (g) | 0.0005 | 0.0000 | 0.5152 |

| Key: |
|---|
| Baseline Solvents |
| * Series 1 |
| ** Series 2 |

# Cleaning Efficiency of Solvents *Continued*

**Discussion**

- 3 potential baseline solvents identified
- Comparison to past test: "WSTF-IR-0134"
  - AK-225: 99% CEF
  - CFC-113: 97% CEF

Limited data on solvents, F,C,G,H,I

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|---|
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# Conclusion

# Conclusion

- The use of AK-225G in the validation step proves additional assurance that candidate solvents qualify as baseline solvents.
- Test results that provide  percent cleaning efficiencies provide guidance into selecting baseline solvents.
- Future studies should consider adequate sample size to better define cleanliness efficiency.

# Questions ?

# Back up slides

# NVR Measured in Solvent Blanks

| Solvent: | Measured NVR (mg) in 100 ml | Comments |
|---|---|---|
| C-HFE-(average) | 3.3 | Sample from Pressure Vessel |
| C-HFE | 0.3 | Sample from new bottle |
| D-HFE | 0.4 | |
| E-Solvent Blend | 106700.3 | Sample from Pressure Vessel |
| E-Solvent Blend | 2.8 | Sample from new mixture |
| F-HFE | 0.1 | Sample from bottle |
| G-HFE | 0.2 | Sample from bottle |
| H-HFE | 0.1 | Sample from bottle |
| I-HFE | 0.3 | Sample from bottle |
| J-Terpene | 3.1 | Sample from bottle. |
| K-HFE | 0.1 | Sample from bottle |

- NVR criteria for solvents needs to be identified early on
- Evaporation rates role in analysis process
- Equipment compatibility with solvent
  - Beaker vs. pressure vessel

# Lessons Learned/ Challenges

- Measure contaminates
- Identifying baseline, cleaning & verification solvent requirements.
- Testing location
  - Clean room
  - Vent hood
- Cross Contamination
- Experiment vs. Cleanroom practices.